L9: Entry 81 of 136 File: USPT Mar 11, 1997

DOCUMENT-IDENTIFIER: US 5610109 A TITLE: Porous ceramic material

### BSPR:

The creation of these metal oxide ceramic materials is generally conducted through a <u>sol-gel</u> procedure. Usually, the metal oxide is initiated into the process as a metal alkoxide solution. The metal is hydrolyzed to metal hydroxide monomers, clusters or particles, depending on the quantity of solvent used. The insoluble metal oxide particles are then peptized by the addition of an acid, or in some cases a base, which causes the particles of the metal oxide to have a greater tendency to remain in suspension, presumably due to charges acquired by the particles during the peptizing process.

#### BSPR:

Two current limitations on the use of ceramic materials are the fabrication materials and the material pore size and distribution range. With regard to the composition of the materials, ceramic materials have been created using many starting materials. For example, Leenaars et al., Jour. of Membrane Science, 24:261-270 (1985), report the use of the sol-gel procedure to prepare supported and unsupported alumina membranes. However, this particular sol-gel process had inherent lower limits as to the size of the particles that could be produced in the sol thus making a lower limit on the size of the pores in the membranes. Ceramic membranes composed of titania, zirconium and other metal oxides have also been reported.

# BSPR:

Alumina membranes have been extensively studied. In particular, Yoldas conducted significant research on the fabrication of gamma-alumina membranes made by a sol-gel process. Yoldas was able to achieve a relatively small particle size in the sols and was able to achieve porous membranes. Yoldas, Jour. Mat. Sci., 12:6, pp. 1203-1208 (1977). In general, however, the method of Yoldas did not permit sufficiently fine control over the sol-gel process to produce uniformly small particles. As a result, the resulting membranes proved less useful for filtration, catalysis or adsorption. For example, in the transparent alumina membranes Yoldas reported that he was able to achieve materials that had small pores, having a radius less than 50 Angstrom, but only in conjunction with the materials which had "larger river-like pores" having a significantly higher radius and contributing significantly to the porosity of the material. Yoldas, Cer. Bull., 54:3, 286-288 (1975).

# BSPR:

Yoldas also investigated the manufacture, through the <u>sol-gel</u> processes, of mixed alumina and silica materials. One class of materials which Yoldas prepared were alumina-siloxane derivatives which formed polymeric cross-linkages making polymeric, rather than particulate, ceramic materials. Yoldas was also able to make several large dense monolithic glass samples of varying compositions of silica and alumina that did retain some porosity and high surface area, as described in the article in Jour. Mat. Sci., supra. Yoldas did not report any ability to make particulate aluminosilicate porous membranes, or the ability to make aluminosilicate membranes having high porosity with exceedingly small pore sizes approaching those of the alumina membranes which he had made.

# BSPR

<u>Sol-gel</u> methods have been employed to fabricate aluminosilicates in several published studies. Most previous authors have applied <u>sol-gel</u> techniques to prepare various aluminosilicates either as dense monoliths or as powders. When

Generate Collection

L7: Entry 4 of 5

File: USPT

Nov 21, 1995

DOCUMENT-IDENTIFIER: US 5468427 A

TITLE: Process for making ultra-fine ceramic particles

# BSPR:

Ultra-fine ceramic particles, with particle sizes of less than about 300 nanometers, are useful for many different purposes. Thus, for example, as is disclosed in U.S. Pat. No. 5,061,682 of Aksay et al., "The use of high temperature superconducting ceramic articles and nonsuperconducting ceramic articles requires reproducible production of the articles from ceramic powders with high densities, high purities, good homogeneity, and fine grain size" (see column 1 of this patent).

### BSPR:

However, the prior art methods for producing ultra-fine ceramic particles are not entirely satisfactory. Thus, as is disclosed in column 1 of this patent, "A common method for producing superconductive ceramic powders involves grinding ceramic powders . . . The grinding produces particles that are greater than one micron in size, are not equiaxed, have a broad particle size distribution, and are often contaminated by the grinding media. Techniques such as <a href="sol-gel">sol-gel</a>, precipitation, and freeze-drying have been developed to overcome some of these undesirable features . . .; however, most of these alternative techniques cannot directly produce superconductive ceramic particles."

### DEPR

By way of illustration, and with reference to the January, 1991 issue of Ceramic Industry (Business News Publishing Company, 755 West Big Beaver Road, Suite 1000, Troy, Mich.), the metal oxide material to be produced by the process may be albite, alumina, aluminum silicate, aluminum titanate, andalusite, antimony oxide, arsenic oxide, barium aluminate, barium oxide, barium titanate, barium tungstate, barium zirconate, bentonite, beryl, beryllium oxide, bismuth stannate, bismuth titanate, bismuth zirconate, borax, cadmium oxide, cadmium zirconate, calcium antimonate, calcium titanate, cerium oxide, chromium oxide, clinochlore, cobalt oxide, copper oxide, cordierite, cristobalite, erbium oxide, europium oxide, forsteite, gadolinium oxide, gallium oxide, germanium dioxide, halloysite, hectorite, holmium oxide, ilmenite, indium oxide, germanium dioxide, halloysite, hectorite, holmium oxide, ilmenite, indium oxide, magnesium oxide, lanthanum oxide, lead oxide, lead zirconate titanate, lutetium oxide, magnesium oxide, manganese dioxide, molybdic oxide, mullite, neodymium oxide, nepheline, nickel oxide, niobium oxide, olivine, petalite, praseodymium oxide, pyrolusite, pyrophyllite, rhodium sesquioxide, samarium oxide, scandium oxide, titania, vanadium pentoxide, ytterbium oxide, mixtures thereof, and the like.

# DEPR:

The second step of the <u>nanoparticle</u> production process provides the ignition of the submicron precursor powders which, in one embodiment, consist of an <u>explosive</u> mixture whose composition is strictly analogous to that of gunpowder. Upon rapid heating, a highly exothermic and rapid chemical reaction occurs that raises the temperature of the particles, causes formation of a target ceramic material to take place, and generates a large amount of gas, leading to nearly instantaneous disintegration of the precursor particles as the nanoparticles are formed.

# DEPR

In one preferred embodiment, not shown, after particle formation by combustion synthesis, the <u>nanoparticles</u> are be coated by a vapor- or liquid-phase deposition technique. In this embodiment, the <u>nanoparticles</u> are placed in a chamber that is filled with coating material vaporized by an evaporization method.

# WEST

# **End of Result Set**

Generate Collection

L9: Entry 136 of 136

File: USPT

Mar 4, 1986

DOCUMENT-IDENTIFIER: US 4574003 A TITLE: Process for improved densification of  $\underline{sol-gel}$  produced alumina-based ceramics

#### ABPL:

The  $\underline{\text{sol-gel}}$  process for forming dense, alumina-base ceramics, the process comprising

#### BSPR:

This invention relates to the production of dense, alumina-based ceramic materials which are useful as, among other things, abrasive grains. In one aspect, this invention relates to producing dense alumina-based ceramics from a sol-qel process while in another aspect, the invention relates to the use of densification aids in the process which facilitate sintering, and thereby improves the densifications of the final product.

#### BSPR

The preparation by a <u>sol-gel</u> process of dense, alumina-based ceramic abrasive grain is known. U.S. Pat. No. 4,314,827 describes a process of making an abrasive mineral employing chemical ceramic technology by gelling a colloidal dispersion or hydrosol of alumina containing at least one precursor of at least one modifying component followed by dehydration and firing. A preferred method of addition of the modifying component is in the form of a soluble salt such as magnesium nitrate. Other disclosures in this area include U.S. Pat. No. 4,181,532 and "Application of <u>Sol-Gel</u> Processes to Industrial Oxides", Jan. 13, 1968, Chemistry and Industry.

# BSPR

While all of these references disclose useful techniques for making dense alumina based ceramic material useful as abrasive grains, none produces a completely satisfactory product. The material which results from most, if not all, of these processes is well below its theoretical density. However, improvement with respect to the density achieved under conventional firing conditions can be made. When used as abrasives, this increased density results in an improvement to their performance in abrasive products. Moreover, most of the <a href="sol-gel">sol-gel</a> processes described in these references can be improved in terms of the source of alumina monohydrates which they process into an acceptable product. Typically acceptable results are achieved when expensive, very high quality alumina monohydrates are used while the use of less expensive materials may require special processing steps.

# BSPR:

The use of various additives to control porosity in alumina-based ceramics produced by a sol-gel process is also known. U.S. Pat. No. 3,417,028 describes a process in which a water-dispersible polyethylene glycol may be incorporated into a hydrous gel of alumina. The mixture is calcined to remove the glycol completely resulting in an inorganic product of increased pore volume and pore size which is useful as a catalyst carrier. Other disclosures in the art include U.S. Pat. No. 2,816,079 (ethylene oxide) and U.S. Pat. No. 3,325,247 (polyethylene glycol). While these references describe useful porous alumina-based ceramics, tney do not suggest that improvement in terms of increased density or reduced open porosity of alumina monohydrate based sol-gel process alumina ceramics may be obtained when sintered at high temperature from the use of these organic additives.

BSPR:

Generate Collection

File: USPT

L9: Entry 112 of 136

Jun 22, 1993

DOCUMENT-IDENTIFIER: US 5222092 A

TITLE: Laser dye impregnated silica sol-gel monoliths

# ABPL:

The present invention relates to a dye laser comprising a highly porous, consolidated silica sol-gel monolith having incorporated therein at least one laser dye, wherein said dye laser is substantially solvent free. The dye laser is prepared by immersing a highly porous, consolidated silica sol-gel monolith in a solution of at least one laser dye and at least one solvent until the solution enters the pores of the monolith to a significant degree to form an impregnated silica sol-gel monolith, and then drying the impregnated silica sol-gel monolith to vaporize substantially all the solvent present within the pores of the monolith.

# BSPR:

Another prior art attempt to solve the above-mentioned problem is disclosed in Avnir, The Nature of the Silica Cage as Reflected by Spectral Changes and Enhanced Photostability of Trapped Rhodamine 6G, J. Phys. Chem., Vol. 88, pp. 5956-5959 (1984). Avnir's article discloses the incorporation of Rhodamine 6G dye into a sol-gel derived silica matrix by adding the Rhodamine 6G dye to a silica sol prior to gellation. When a dopant compound is mixed into a sol before gellation, however, gradients are inevitably formed in the final product due to the migration of the dopant to the surface of the product during the subsequent aging and drying stages. Reaction byproducts are thus trapped within the matrix. Further, dye lasers prepared according to this method cannot be subjected to high temperature stabilization treatments without risking the decomposition of the incorporated Rhodamine 6G dye.

# BSPR:

The present invention, which solves the problems of the prior art, relates to a dye laser comprising a highly porous, consolidated silica sol-gel monolith having incorporated therein at least one laser dye, wherein said dye laser is substantially solvent free.

# BSPR:

(e) stabilizing said dried, aged gel to form a highly porous, consolidated silica sol-gel monolith; and

# BSPR:

(f) immersing said highly porous, consolidated silica <u>sol-gel</u> monolith in a solution comprising at least one laser dye and at least one solvent until the solution enters the pores of the monolith to a significant degree to form a laser dye impregnated silica <u>sol-gel</u> monolith; and

# BSPR

(g) drying said impregnated silica <u>sol-gel</u> monolith to vaporize substantially all the solvent present within the pores of said monolith to form a substantially solvent free laser dye impregnated silica\_sol-gel monolith.

# BSPR

The present invention further relates to a tunable lasing system having a laser cavity comprising (a) a dye laser medium comprising a highly porous, consolidated silica sol-gel monolith having incorporated therein at least one laser dye, wherein said dye laser medium is substantially solvent free, and (b) an optical pumping excitation means, such as a laser beam, operably coupled with said dye laser medium. The tunable lasing system can be operated with a pulsed or

#### DEPR

In one preferred embodiment, a sol gel process is used to coat the combusted powder. The precursor to the <u>sol-gel</u> coating material can be a metal alkoxide. These alkoxides are easily hydrolyzed, and the network-forming elements (silicon, boron, titanium, and the like) can be polymerized to form a gel rather than a precipitate. The metal hydroxide is first hydrolyzed, replacing the OR.sup.- with OH.sup.- in the M(OR).sub.n metal alkoxide. The metal hydroxide is then condensed to form monomers ([MO].sup.-) which combine to form trimers, chains, rings, etc. and finally link to form a three-dimensional network.

### DEPR:

In one preferred embodiment, the combusted powder is ball-milled in a  $\underbrace{\text{sol-gel}}$  forming solution containing from about 0.5 to about 5.0 mole percent of tetraethyl orthosilicate in deionized water. The dispersed slurries are dried by evaporation at 40 degrees centigrade with constant stirring to avoid segregation.

# DEPR:

Deposition of uniform <u>sol-gel</u> coatings can aid in the ability to control the surface charge of the <u>particles</u>, allow for faster rearrangement of particles during the first stages of liquid phase sintering, reduce the time required for complete sintering, and aid in the homogenization of the distribution of dopants around the particles.

# CLPR:

in an  $\underline{\text{explosive}}$  manner. 2. The process as recited in claim 1, further comprising the steps of:

# CLPR:

16. The process as recited in claim 1, wherein said combusted powder is milled in a sol-gel forming solution.

# CLPR:

17. The process as recited in claim 16, wherein said  $\underline{sol-gel}$  forming solution is comprised of tetraethyl orthosilicate.

# WEST

Generate Collection

L9: Entry 95 of 136

File: USPT

Nov 21, 1995

DOCUMENT-IDENTIFIER: US 5468427 A

TITLE: Process for making ultra-fine ceramic particles

### BSPR:

However, the prior art methods for producing ultra-fine ceramic particles are not entirely satisfactory. Thus, as is disclosed in column 1 of this patent, "A common method for producing superconductive ceramic powders involves grinding ceramic powders . . . The grinding produces particles that are greater than one micron in size, are not equiaxed, have a broad particle size distribution, and are often contaminated by the grinding media. Techniques such as <a href="sol-gel">sol-gel</a>, precipitation, and freeze-drying have been developed to overcome some of these undesirable features . . .; however, most of these alternative techniques cannot directly produce superconductive ceramic particles."

#### DEPR:

The cation of the metal(s) used in the mixture may be any cation which can be incorporated in a compound which is capable of being converted into a metal oxide by heating. By way of illustration and not limitation, one may use the nitrate, the perchlorate, the sulfate, the carbonate, the chloride, the oxalate, the acetate, the hydroxide, and the like as the anion of the metal cation-containing compound, provided that the metal cation compound preferably is soluble in the solvent used in the process.

### DEPR:

In one preferred embodiment, a sol gel process is used to coat the combusted powder. The precursor to the <u>sol-gel</u> coating material can be a metal alkoxide. These alkoxides are easily hydrolyzed, and the network-forming elements (silicon, boron, titanium, and the like) can be polymerized to form a gel rather than a precipitate. The metal hydroxide is first hydrolyzed, replacing the OR.sup.- with OH.sup.- in the M(OR).sub.n metal alkoxide. The metal hydroxide is then condensed to form monomers ([MO].sup.-) which combine to form trimers, chains, rings, etc. and finally link to form a three-dimensional network.

# DEPR:

In one preferred embodiment, the combusted powder is ball-milled in a  $\underline{\text{sol-gel}}$  forming solution containing from about 0.5 to about 5.0 mole percent of tetraethyl orthosilicate in deionized water. The dispersed slurries are dried by evaporation at 40 degrees centigrade with constant stirring to avoid segregation.

# DEPR:

Deposition of uniform  $\underline{\text{sol-gel}}$  coatings can aid in the ability to control the surface charge of the  $\overline{\text{particles}}$ , allow for faster rearrangement of particles during the first stages of liquid phase sintering, reduce the time required for complete sintering, and aid in the homogenization of the distribution of dopants around the particles.

# CLPR:

16. The process as recited in claim 1, wherein said combusted powder is milled in a  $\underline{sol-gel}$  forming solution.

# CLPR:

17. The process as recited in claim 16, wherein said <u>sol-gel</u> forming solution is comprised of tetraethyl orthosilicate.

L7: Entry 2 of 5

File: USPT

May 11, 1999

DOCUMENT-IDENTIFIER: US 5902569 A TITLE: Ultraviolet shielding composite fine particles, method for producing the same, and cosmetics

#### DEPR:

In the preparation of the liquid mixture in step (a) above, the powders of the daughter particles where these particles comprise ultrafine particles having an average particle diameter of from 0.001 to 0.1 .mu.m are desirably disintegrated and/or pulverized in a mill or an apparatus for high-pressure dispersion, whereby the dispersion state of the daughter particles in the liquid mixture is maintained. Examples of mills include bead mills, sand mills, and ball mills, and examples of high-pressure dispersion devices include microfluidizers and nanomizers.

### DEPR:

In the coating step with the inorganic materials according to step (b), the coating methods are not particularly limited, and any of the coating methods utilized for sol-gel reaction methods and precipitation reaction methods can be employed. The inorganic materials to be used for coating are not particularly limited as long as they are inorganic materials having substantially no catalytic activity. Among them, a preference is given to metal oxides, with a particular preference being given to SiO.sub.2, Al.sub.2 O.sub.3, and a mixture thereof. The starting materials for the inorganic materials used in the coating step are not particularly limited, and any of the metal salts, such as metal alkoxides, metal nitrates, and metal sulfates, may be used. Specific examples of the metal salts include tetraethoxysilane, aluminum isopropoxide, aluminum tri-sec-butoxide, sodium silicates, and aluminum sulfate. By utilizing the precipitation reaction and the sol-gel reaction depending upon the properties of the above starting materials for the inorganic materials used, the surface of the composite fine particles can be coated with the inorganic materials. Incidentally, after the coating step, a neutralization reaction step may be optionally added before or after the water-repellent treatment step.

# DEPR:

In the present specification, the "sol-gel reaction method" refers to a method comprising allowing colloidal particles contained in a sol to grow by coagulation or aggregation, thereby allowing gelation of the resulting colloidal particles to take place. In the case where the above sol-gel reaction method is utilized to coat the surface of the composite fine particles, the gelation mentioned above can be carried out on the surface of the composite fine particles. Also, the "precipitation reaction method" refers to a method comprising adding a precipitation agent to a solution containing the metal salts, to give precipitated particles. In this method, concentrations, pHs, and temperatures are suitably controlled to have the precipitated particles adhere on the surface of the composite fine particles. Examples of the precipitation reaction methods include coprecipitation methods, methods of precipitation from homogeneous solution, and compound precipitation methods.

# DEPR

Thereafter, the procedure of dispersing the starting material liquid mixture thus prepared using a high-pressure dispersing device ("LA-31," manufactured by Nanomizer INC.) was repeated ten times under the disintegration pressure of 1000 kg/cm.sup.2. By the above dispersion treatment, a liquid dispersion containing ZnO/SiO.sub.2 composite fine particles was obtained.

DEPL:

(3) Inorganic white pigments such as titanium oxide and zinc oxide; inorganic recipies such as iron oxide (red oxide) and iron titanate; inorganic brown pigments such as igamma.—iron oxide; inorganic yellow pigments such as yellow iron oxide and yellow ochre; inorganic black pigments such as black iron oxide and carbon black; inorganic violet pigments such as manganese violet and cobalt violet; inorganic green pigments such as chromium oxide, chromium hydroxide, and cobalt titanate; inorganic blue pigments such as ultramarine and Prussian blue; pearl-like pigments such as mica coated with titanium oxide, oxychlorobismuth coated with titanium oxide, oxychlorobismuth, talc coated with titanium oxide, fish scale flake, mica coated with colored titanium oxide; and metal powder pigments such as aluminum powders and copper powders.					
DETL:					
Amount Ingredients (weight %)					
Triclosan 0.01 Aluminum hydroxychloride 1.5 Talc 1.0 Composite Fine Particles (Produced 5.0 in Example 2) Isopropyl myristate 2.0 Perfume 0.2 Propellant Balance					
DETL:					
Amount Ingredients (weight %)					
(1) Composite Fine Particles (Produced 10.0 in Example 3) (2) Fluorine Compound-Treated (*1) Mica Balance (3) Fluorine Compound-Treated (*1) Talc 20.0 (4) Fluorine Compound-Treated (*1) 8.0 Titanium Oxide (5) Fluorine Compound-Treated (*1) Iron Oxide 3.0 (Red, Yellow, Black) (6) Fluorine Compound-Treated (*1) Zinc Oxide 2.0 Fine Particles (7) Fluorine Compound-Treated (*1) 1.0 Titanium Oxide Fine Particles (Average, Particle Diameter: 35 nm) (8) Fluorine Compound-Treated (*1) 10.0 Nylon Powder (9) Methyl polysiloxane (10 cSt) 4.0 (10) Perfluoropolyether ("FOMBLIN HC-04," 8.0 manufactured by AUSIMONT CO.) (11) Hydrogenated Oil (Synchrowax) 1.0 (12) Octyl methoxycinnamate 1.0 (13) Antiseptics, Perfume 1.0  Note (*1): Treatment was carried out by coating with 2% by weight of perfluoroalkyl ethyl phosphate.					
DETL: Amount Ingredients (weight %)					
(1) Composite Fine Particles (Produced 20.0 in Example 2) (2) Silicone-Treated (*2) Mica Balance (3) Silicone-Treated (*2) Talc 20.0 (4) Silicone-Treated (*2) Titanium Oxide 9.0 (5) Silicone-Treated (*2) Iron Oxide 4.0 Red, Yellow, Black) (6) Silicone-Treated (*2) Zinc Oxide 8.0 Fine Particles Coated with 30% by weight of Nylon Powder (7) Methyl polysiloxane (10,000 cSt) 0.2 (8) Methyl polysiloxane (6 cSt) 8.0 (9) Hydrogenated Oil (Synchrowax) 1.0 (10) Octyl methoxycinnamate 2.0 (11) Antiseptics, Perfume 1.0 Note (*2): Treatment was carried out by coating with 2% by weight of methylhydrogenpolysiloxane.					
DETL:					
Amount Ingredients (weight %)					
(1) Dispersion Oil Containing Composite  11.0 Fine Particles (Produced in Example 5) (2) Lecithin-Treated (*3) Mica Balance (3) Lecithin-Treated (*3) Titanated Mica 6.0 (4) Silicone-Treated (*4) Ultramarine 8.0 (5) Silicone-Treated (*4) Prussian blue 10.0 (6) Silicone-Treated (*4) Iron Oxide 2.0 (Red, Yellow, Black) (7) Spherical Silicone Resin Powder 10.0 ("TOSPEARL 145," manufactured by Toshiba Silicone Corpation) (8) Diisostearyl malate 3.0 (9) Hydrogenated Oil (Synchrowax) 0.5 (10) Vaseline 1.0 (11) Antiseptics, Perfume 1.0  Treatment was carried out by coating with 5% by weight of soybean lecithin. (*4) Treatment was carried out by coating with 2% by weight of					
Treatment was carried out by coating with 25 by weight of					

DETL:

methylhydrogenpolysiloxane.

Amount Ingredients (weight %)

(1) Dispersion Oil Containing Composite

25.0 Fine Particles (Produced in Example 7) (2) Silicone-Treated (\*5) Titanium

Oxide 3.0 (3) Silicone-Treated (\*5) Iron Oxide 1.5 (Red, Yellow, Black) (4)

Silicone-Treated (\*5) Zinc Oxide 3.0 Fine Particles (5) Dimethylcyclopolysiloxane

10.0 (6) Octyl methoxycinnamate 2.0 (7) Dimethylsiloxane-methylpolyoxyethylene
1.0 siloxane copolymer (8) Glycerol 2.0 (9) Ethanol 10.0 (10) Distilled water

Balance \_\_\_\_\_\_\_\_ Notes (\*5): Treatment was carried

out by coating with 2% by weight of methylhydrogenpolysiloxane.

DETL:

Amount Ingredients (weight %)

(1) Dispersion Oil Containing Composite

25.0 Fine Particles (Produced in Example 13) (2) Silicone-Treated (\*7) Titanium

Oxide 3.0 (3) Silicone-Treated (\*7) Iron Oxide 1.5 (Red, Yellow, Black) (4)

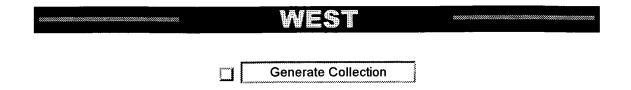
Silicone-Treated (\*7) Zinc Oxide 3.0 Fine Particles (5) Dimethylcyclopolysiloxane

10.0 (6) Octyl methoxycinnamate 2.0 (7) Dimethylsiloxane-methylpolyoxyethylene
1.0 siloxane copolymer (8) Glycerol 2.0 (9) Ethanol 10.0 (10) Distilled water

Balance

Notes (\*7): Treatment was carried out by coating with 2% by weight of methylhydrogenpolysiloxane.

3 of 3



L9: Entry 44 of 136 File: USPT Jan 12, 1999

DOCUMENT-IDENTIFIER: US 5858813 A

TITLE: Chemical mechanical polishing slurry for metal layers and films

#### DEPR:

Precipitated abrasives may be manufactured utilizing conventional techniques and are typically formed by the coagulation of the desired particles from an aqueous medium under the influence of high salt concentrations, acids or other coagulants. The particles are filtered, washed, dried and separated from residues of other reaction products by conventional techniques known to those skilled in the art. In addition, the abrasive may be produced from other suitable technologies, such as sol-gel and plasma processing.

### DEPR:

The oxidizing agent of the present invention is added to the polishing slurry to oxidize the metal layer to its corresponding oxide or ions. For example, in the present invention, an oxidizing agent may be used to oxidize a metal layer to its corresponding oxide, such as aluminum to aluminum oxide or copper to copper oxide. The layer is mechanically polished to remove the respective oxide from the layer. Although a wide range of oxidizing agents may be used, suitable agents include oxidizing metal salts, oxidizing metal complexes, nonmetallic oxidizing acids such as peracetic and periodic acids, iron salts such as nitrates, sulfates, EDTA, citrates, potassium ferricyanide and the like, aluminum salts, sodium salts, potassium salts, ammonium salts, quaternary ammonium salts, phosphonium salts, or other cationic salts of peroxides, chlorates, perchlorates, nitrates, permanganates, persulfates and mixtures thereof. Furthermore, it is expected that water may also be used as an effective oxidizing agent in slurries when electronegative metals are used, such as aluminum. The standard electrochemical potential for the oxidation of, for example, aluminum to Al.sup.+3 -ions is: E.sub.0 =-1.663+0.0197  $\log(Al.sup.+3)$  and for the oxidation to Al.sub.2 O.sub.3, E.sub.0 =-1.550-0.0591 pH, expressed in Volts, V, against normal hydrogen electrode, NHE (as defined in "Atlas of Elechtrochemical Equilibria in Aqueous Solutions" by M. Pourbaix, Pergamon Press, New York, 1966). The standard potential for H.sub.3 O.sup.+ and H.sub.2 O reduction is 0 V on the same scale. The actual potential which can be measured during the abrasion of aluminum in some of the electrolytes, without the addition of oxidizers, is -1.4 V vs. NHE. This potential is low enough for vigorous reduction of both H.sub.3 O.sup.+ and H.sub.2 O. Electrochemical measurements indicate that the prevailing reduction reaction is that of water.

# CLPR:

23. The method of claim 1, wherein said oxidizing agent is selected from the group consisting of: iron salts, aluminum salts, sodium salts, potassium salts, ammonium salts, quaternary ammonium salts, phosphonium salts, peroxides, chlorates, perchlorates, nitrates, permanganates, persulfates and mixtures thereof.

# CLPR:

49. The slurry of claim 32, wherein said oxidizing agent is selected from the group consisting of: iron salts, aluminum salts, sodium salts, potassium salts, ammonium salts, quaternary ammonium salts, phosphonium salts, peroxides, chlorates, perchlorates, nitrates, permanganates, persulfates and mixtures thereof.

L9: Entry 64 of 136

File: USPT

Feb 10, 1998

DOCUMENT-IDENTIFIER: US 5716565 A

TITLE: Process for making ultra-fine stabilized zirconia particles

### DEPR:

The cation of the metal(s) used in the mixture may be any cation which can be incorporated in a compound which is capable of being converted into a metal oxide by heating. By way of illustration and not limitation, one may use the nitrate, the perchlorate, the sulfate, the carbonate, the chloride, the oxalate, the acetate, the hydroxide, and the like as the anion of the metal cation-containing compound, provided that the metal cation compound preferably is soluble in the solvent used in the process.

#### DEPR

In one preferred embodiment, a  $\underline{sol-gel}$  process is used to coat the combusted powder. The precursor to the  $\underline{sol-gel}$  coating material can be a metal alkoxide. These alkoxides are easily hydrolyzed, and the network-forming elements (silicon, boron, titanium, and the like) can be polymerized to form a gel rather than a precipitate. The metal hydroxide is first hydrolyzed, replacing the OR.sup.- with OH.sup.- in the M(OR).sub.n metal alkoxide. The metal hydroxide is then condensed loll to form monomers ([MO].sup.-) which combine to form trimers, chains, rings, etc. and finally link to form a three-dimensional network.

# DEPR:

In one preferred embodiment, the combusted powder is ball-milled in a <u>sol-gel</u> forming solution containing from about 0.5 to about 5.0 mole percent of tetraethyl orthosilicate in deionized water. The dispersed slurries are dried by evaporation at 40 degrees centigrade with constant stirring to avoid segregation.

# DEPR:

Deposition of uniform  $\underline{\text{sol-gel}}$  coatings can aid in the ability to control the surface charge of the  $\overline{\text{particles}}$ , allow for faster rearrangement of particles during the first stages of liquid-phase sintering, reduce the time required for complete sintering, and aid in the homogenization of the distribution of dopants around the particles.

unuu		WES					
Help Logout Interrupt							
Me	ain Menu   Search Form	Posting Counts Show	v S Numbers   Ed	it S Numbers   Preferences			
Search Results -							
		Terms	Documents				
		11 and ((149/\$)!.CCLS	S.) 2				
Database:	US Patents Full Text of JPO Abstracts Databate EPO Abstracts Databate Derwent World Patent IBM Technical Disclos	ase ase ts Index					
Refine Search: Clear							
Search History							

Today's Date: 11/29/2000

<b>DB Name</b>	Query	Hit Count	Set Name
USPT	11 and ((149/\$)!.CCLS.)	2	<u>L7</u>
USPT	15 and (explosive or propellant or energetic)	5	<u>L6</u>
USPT	13 or 14	102	<u>L5</u>
USPT	12 and (nano\$)	102	<u>L4</u>
USPT	12 and (nano\$)	98	<u>L3</u>
USPT	11 and iron adj oxide	361	<u>L2</u>
USPT	sol-gel	4594	<u>L1</u>